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(54) Title: PRESSURE AND TEMPERATURE SWING ADSORPTION AND TEMPERATURE SWING ADSORPTION

(57) Abstract

A method of heating a pressure and temperature swing adsorption gas filtration bed unit is provided comprising locating a heating means upstream of the adsorbent material within the filtration bed characterised in that the heating means acts to heat gas passing into the bed or a layer thereof in the purge direction and uses the heated air to heat the adsorbent material.

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PRESSURE AND TEMPERATURE SWING ADSORPTION AND TEMPERATURE
SWING ADSORPTION.

The present invention relates to a method of separating components from a gaseous mixture by use of temperature swing adsorption and, more particularly, pressure and temperature swing adsorption, and to an improved method and apparatus for enabling regeneration of adsorbent materials in one or more beds for use in gas filtration. Particularly the present invention is concerned with filtering volatile contaminants from oxygen and/or nitrogen containing gas mixtures such as air.

Pressure swing adsorption processes are suited to air separations involving light gases, but not those involving high boiling point components which are strongly adsorbed to the filter bed (see applicant's copending application reference UK 94 22833.5). This limitation can be overcome by using a combination of pressure and temperature swing, as provided using a pressure and temperature swing adsorber (PTSA) bed, using an appropriate adsorbent inventory, where the temperature swing serves to desorb the high boiling point components.

The use of a combined pressure and temperature adsorption stage will minimise the size of the filter bed. A temperature swing adsorption system is however feasible when power and other limitations prevent the use of a compressor device. A filter bed based on temperature swing adsorption (TSA) alone will however be larger than one based on a combination of pressure and temperature.

The present inventors have determined that heating systems to be employed in a PTSA bed or TSA bed for use in such method should possess most or all of the following characteristics: compactness to allow minimal size of adsorber bed; non-intrusiveness in the bed interior giving it the ability to be snow-storm filled; axial positioning to enable layered adsorbents to be used in a single bed whilst preventing fluidisation; intrinsic safety for prevention of bed overheating and thermal ageing; the ability to heat and cool in consistent and reproducible manner to give long term stability;

low thermal mass allowing rapid and efficient heating and cooling; efficient and homogeneous axial and radial. transfer to minimise regeneration period such as to generate a rectangular heating and cooling profile; and economical power consumption allowing flexible deployment of the system in mobile equipments.

There are a number of methods available for generating the temperature swing but many of these suffer from one or more limitations. Examples of methods excluded from practical study include microwave systems and direct electrical heating of the adsorber bed.

Microwave systems would require a generator and waveguide and would heat the bed in a highly localised fashion. Materials of construction, including the adsorber beds and the adsorbents would be limited by the constraints associated with the use of microwaves resulting in the risk of thermal degradation and combustion of the adsorbents being high. Heat transfer within the bed would rely on interparticle conduction and would therefore be inefficient.

Direct electrical heating would suffer from some of the above limitations including combustion hazards associated with the use of live electrical currents, including current leakage, and the need for stringent electrical isolation of the adsorber beds would increase the complexity of the equipment. In addition the use of this system would limit the choice of adsorbent.

Although both techniques are non-intrusive their use would constrain adsorbent choice. The present inventors have now provided a method for heating such PTSA beds that allows use of commercially available heaters and does not limit the adsorbent in this manner.

Thus in a first aspect of the present invention there is provided a method of heating a pressure and temperature swing adsorption gas filtration bed unit, or temperature swing adsorption gas filtration bed unit, comprising locating a heating means within the bed housing characterised in that the heating means acts to heat gas passing into the bed or a layer thereof in the purge direction and uses the heated air to heat the adsorbent material.

The purge direction will be understood to be the direction of gas flow used when the bed is being regenerated and will conventionally be counter to the flow direction when the bed is being used to separate contaminant or other components desired to be separated from a gas to be treated.

In a preferred embodiment of this aspect of the present invention the method employs a number of layers of adsorbent material within the filtration bed housing and positions a heating means upstream, with respect to the purge flow direction, of each of these layers. Most preferably the heating means are used to separate different adsorbent layers positioned within a single pressure and temperature swing adsorber bed unit, or temperature swing adsorber bed unit, such as the multilayer bed units that are the subject matter of the applicant's copending application UK 94 22833.5).

In this manner the temperature selected for regeneration of a particular adsorbent material may be matched more closely to its particular characteristics, particularly with regard to its thermal degradation characteristics and the amount of heat required to purge a particular component from a particular layer at a given pressure.

Particularly preferred heaters for the purpose of heating the gas as it enters each layer of a bed are provided in the form of disc shaped heater units located within divider elements which may be used for supporting and in turn being supported by the adsorbent of an adjacent adsorbent material layers. Suitable such heater units are Curie point heaters such as those provided by Domnick Hunter Filters UK, (these being conveniently located in batteries of three heater elements each) or any other arrangement including batteries containing six or more such discs, or elements of different shape.

The heaters are controlled, in the preferred design, using a microprocessor device (there being no need to monitor bed temperature). The microprocessor device allows rapid control of the bed heaters including the provision of sequential shutdown in order to minimise cooling periods required and thus return the bed to operational condition as soon as possible.

The electrical connections to the heater batteries and sensors may conveniently be provided entering through the ends of the bed, as will be seen in the Example below. The electrical connections can also be made through the walls of the housing, the housing may also be of different configuration, eg circular, and may use a different heater battery design containing six or more curie point elements, which themselves may be of different shape.

The particular use of batteries of air heaters placed within transverse elements to divide a bed into layers allows placement of heaters at any desired position within the bed. The preferred Curie point heaters are of honeycomb construction and provide direct gas heating during passage of gas through the bed with gas temperature controlled by the composition of the heater element.

The present invention will now be described further by way of illustration only by reference to the following non-limiting Figures and Example. Further embodiments falling within the scope of the claims will occur to those skilled in art in the light of these.

FIGURES.

Figure 1: shows a section view of (i) a conductive coil heated bed and (ii) the coil used as a comparative example.

Figure 2: shows temperature profiles obtained using the coil heater of Figure 1 from thermocouples placed in the bed core or adjacent the housing wall.

Figure 3: shows (i) elevation section and cross section (ii) through a comparative example rod and vane heated bed.

Figure 4: shows temperature profiles obtained using the rod and vane heater of Figure 2 obtained from lower manifold temperature.

Figure 5a shows a plan view and an elevation of a 3 element Curie point heater showing its dimensions. Figure 5b shows a divider element in which the heater of figure 5a is placed during operation.

Figure 6 shows the arrangement of four heater units of Figure 5 within a vertically oriented bed such as to divide it into four layers.

Figure 7 shows temperature profiles obtained within a bed of zeolite using a 38.5 dm^3 fill at airflow rate $3120 \text{ dm}^3 \text{ min}^{-1}$ at the thermocouples A1 to A6 of Figure 6.

EXAMPLE 1: Curie point air heater pressure and temperature swing adsorber unit.

Curie point heaters (three elements per battery, see figure 5) and housings were supplied by Domnick Hunter Filters Ltd, UK. Referring to figure 6, four batteries 1 - 4, each independently controlled via a microprocessor keypad timing device, were located axially within a 38.5 dm^3 bed of zeolite, electrical connection being made by thin (2mm) insulated wires which passed along the adsorber bed to connectors introduced into an upper manifold via gas tight ducts. The upper and lower manifolds, which formed an integral part of the bed housing, also contained the valving arrangement. Each element was of honeycomb construction and provided direct air heating during passage of gas along the column of the bed unit. Gas temperature was controlled by the composition of the heating element and was approximately $180\text{-}210^\circ\text{C}$. Power consumption was approximately 2kW (eight three element blocks occupying 10% of the housing volume). Typical in-bed temperature profiles during heating and cooling measured using thermocouples shown at positions A1 to A6 are shown in Figure 7 with a gas flow of air at $3120 \text{ dm}^3 \text{ min}^{-1}$. In obtaining these the heater batteries were switched off sequentially.

Countercurrent purge flow applied during regeneration was varied with higher flowrates reducing bed temperatures and thus causing more power to be consumed. The temperature profile for the Curie point air heater bed of the invention should be compared with those obtained using a conductive coil heater (Figure 1) and a rod and vane heater (Figure 3).

The conductive coil heated bed used a conductive coil electrically connected through the bed housing base with a thermocouple placed close to the element. The element occupied 13% of the housing interior volume and the heater provided 0.6kW to the adsorbent variable by increasing or decreasing applied current. The valving arrangement for process control was positioned remote from the housing and typical in-bed temperatures measured using thermocouples placed radially and axially are shown in Figure 2 (0.5 dm^3 activated carbon fill product airflow $55 \text{ dm}^3 \text{ min}^{-1}$).

The conductive rod and vane heaters and housings utilise vane heating via an electric rod inserted centrally along the bed length with temperature control achieved via a remote current control device. Power consumption did not exceed 4.4kW. The rod and vane arrangement occupied approx. 22% of the housing volume. Typical in-bed temperature profiles during heating and cooling obtained using thermocouples at sampling points SP0-SP5 shown in Figure 3 are shown in Figure 4 (6.8 dm^3 zeolite fill, airflow rate $1400 \text{ dm}^3 \text{ min}^{-1}$). The valving arrangement for process control was positioned remote from the housing.

CLAIMS.

1. A method of heating a pressure and temperature swing adsorption gas filtration bed unit, or temperature swing adsorption gas filtration bed unit, comprising locating a heating means within the filtration bed housing characterised in that the heating means acts to heat gas passing into the bed or a layer thereof in the purge direction and the heated air is used to heat the adsorbent material.
2. A method as claimed in claim 1 wherein the direction of gas flow used when the bed is being regenerated is counter to the flow direction when the bed is being used to separate contaminant or other components desired to be separated from a gas to be treated.
3. A method as claimed in claim 1 or claim 2 wherein a number of layers of adsorbent material are employed within the filtration bed unit with a heating means positioned upstream, with respect to the purge flow direction, of two or more of these layers.
4. A method as claimed in any one of the preceding claims wherein the heating means are used to separate different adsorbent layers positioned within a single pressure and temperature swing adsorber bed unit or temperature swing adsorber bed unit.
5. A method as claimed in any one of the preceding claims wherein the heating means comprise heater units located within divider elements which may be used for supporting and in turn being supported by the adsorbent of an adjacent adsorbent material layer.
6. A method as claimed in claim 5 wherein the heating means comprise Curie point heaters.
7. A pressure and temperature swing adsorption filtration bed unit, or temperature swing adsorption filtration bed unit, comprising a heating means located upstream of an adsorbent material within the filtration bed housing in the purge direction characterised in that the heating means acts to heat gas passing into the bed or a layer thereof in the purge direction and uses the heated air to heat the adsorbent material.

Fig.1a.

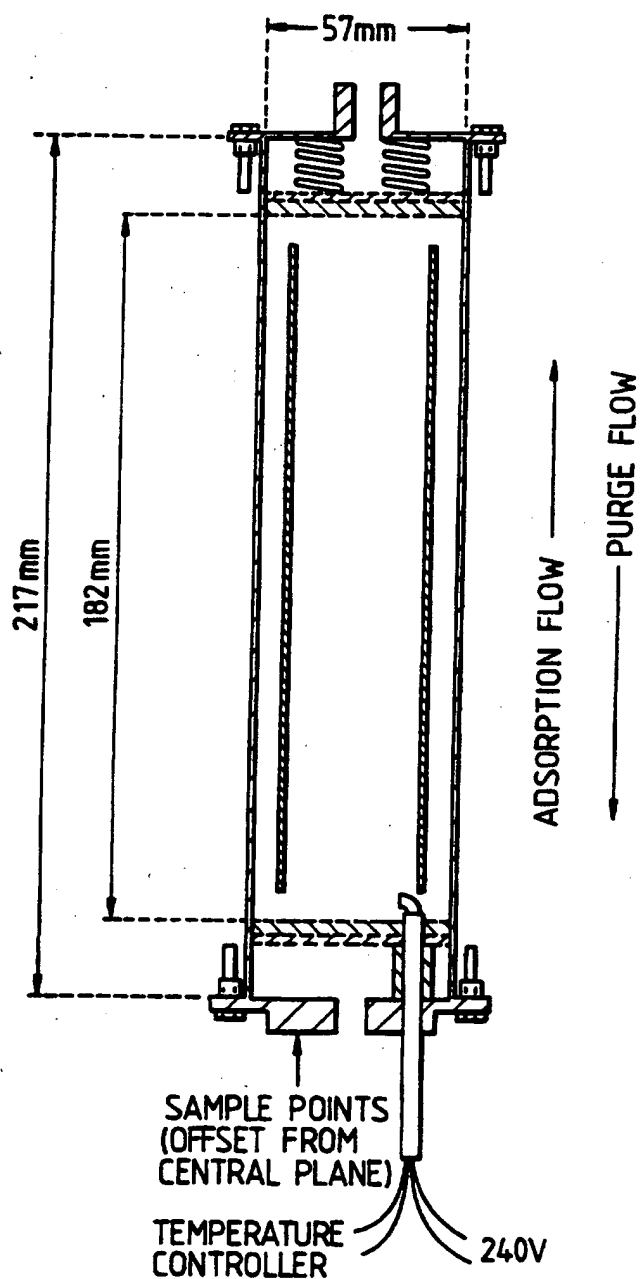
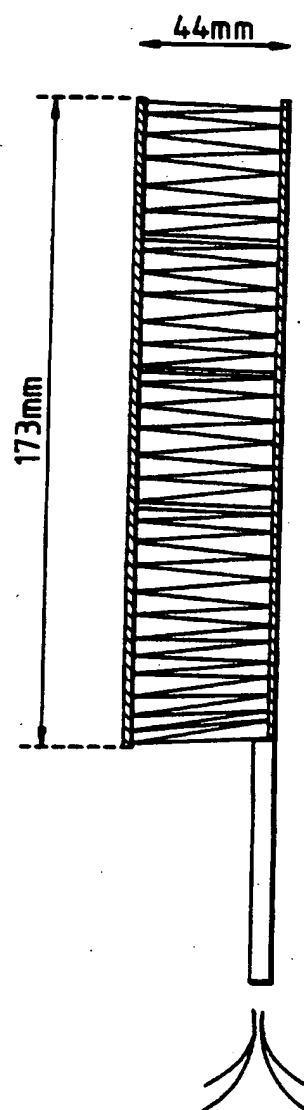


Fig.1b.



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Fig.2.

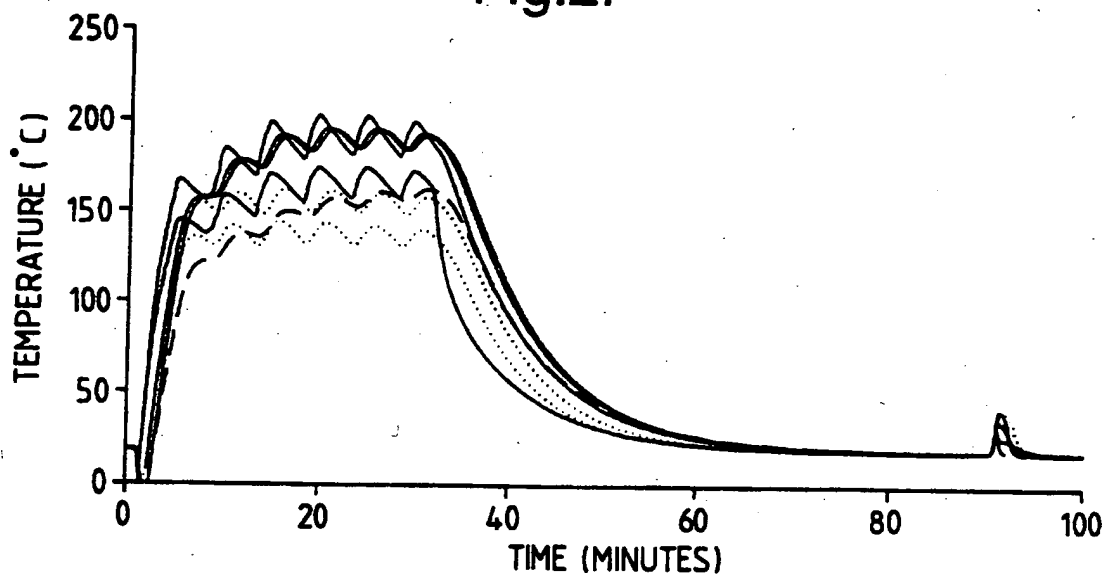


Fig.4.

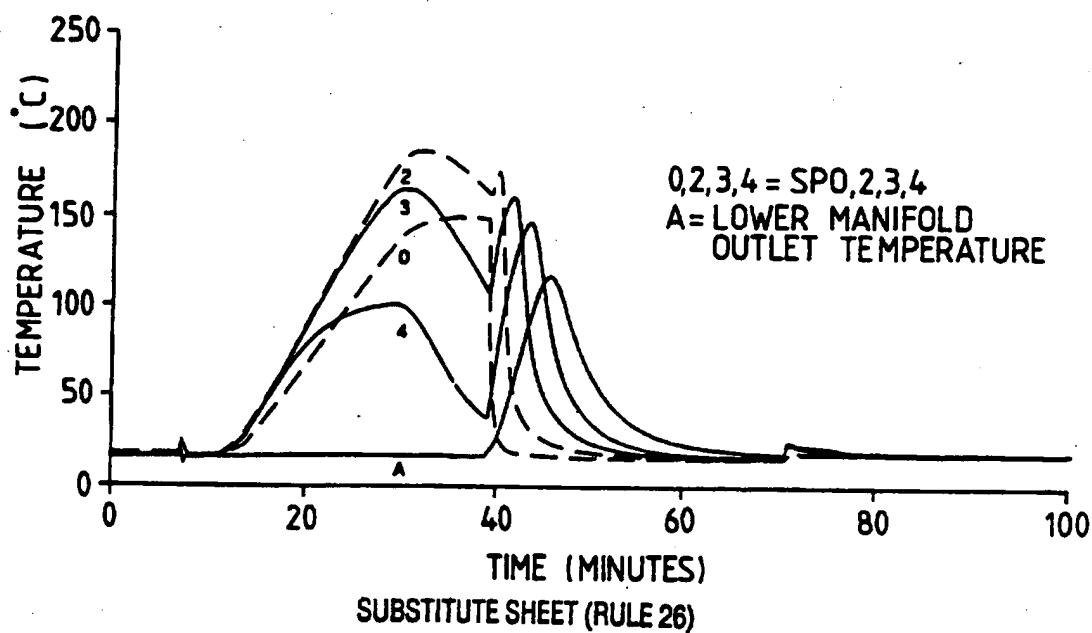
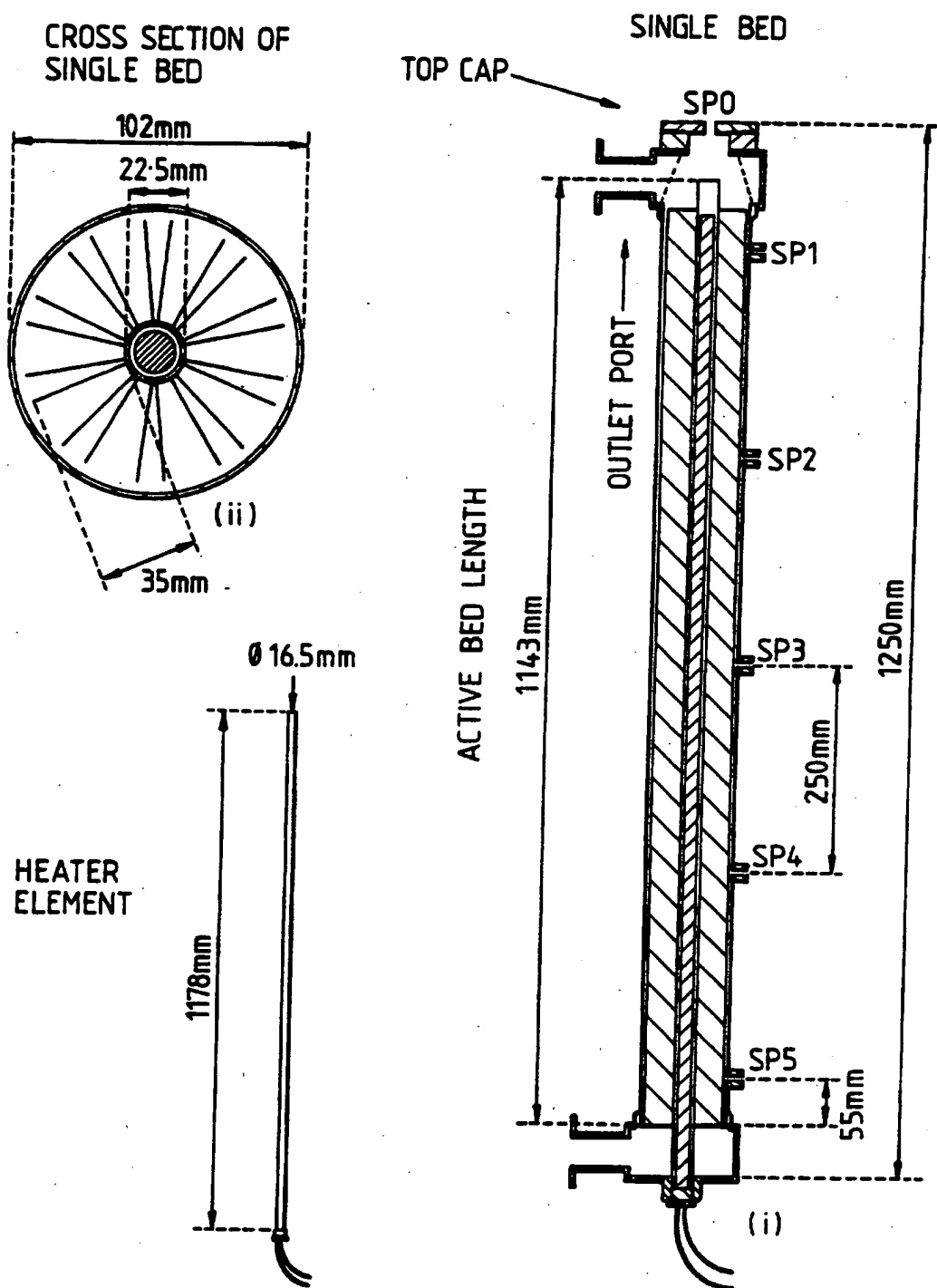


Fig.3.



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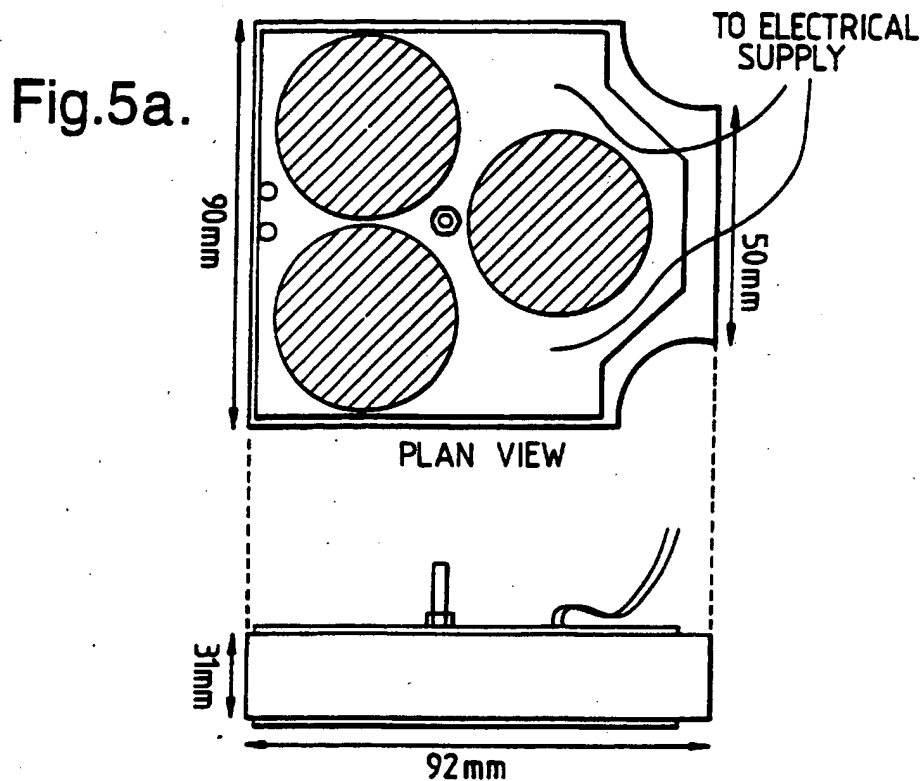
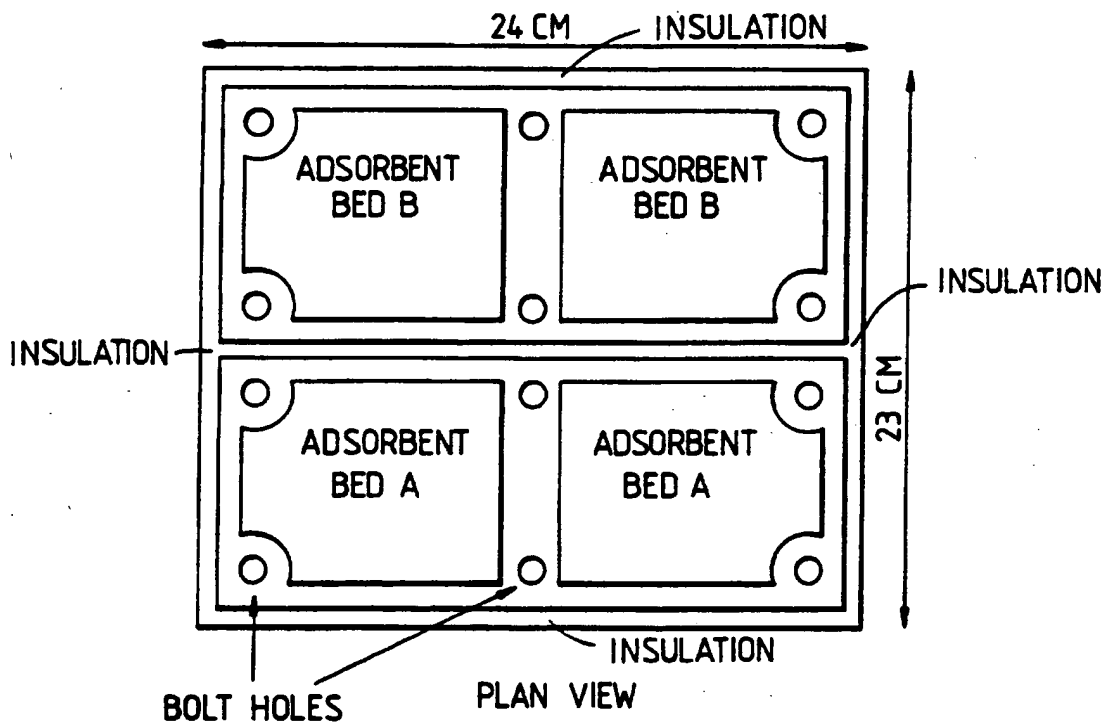
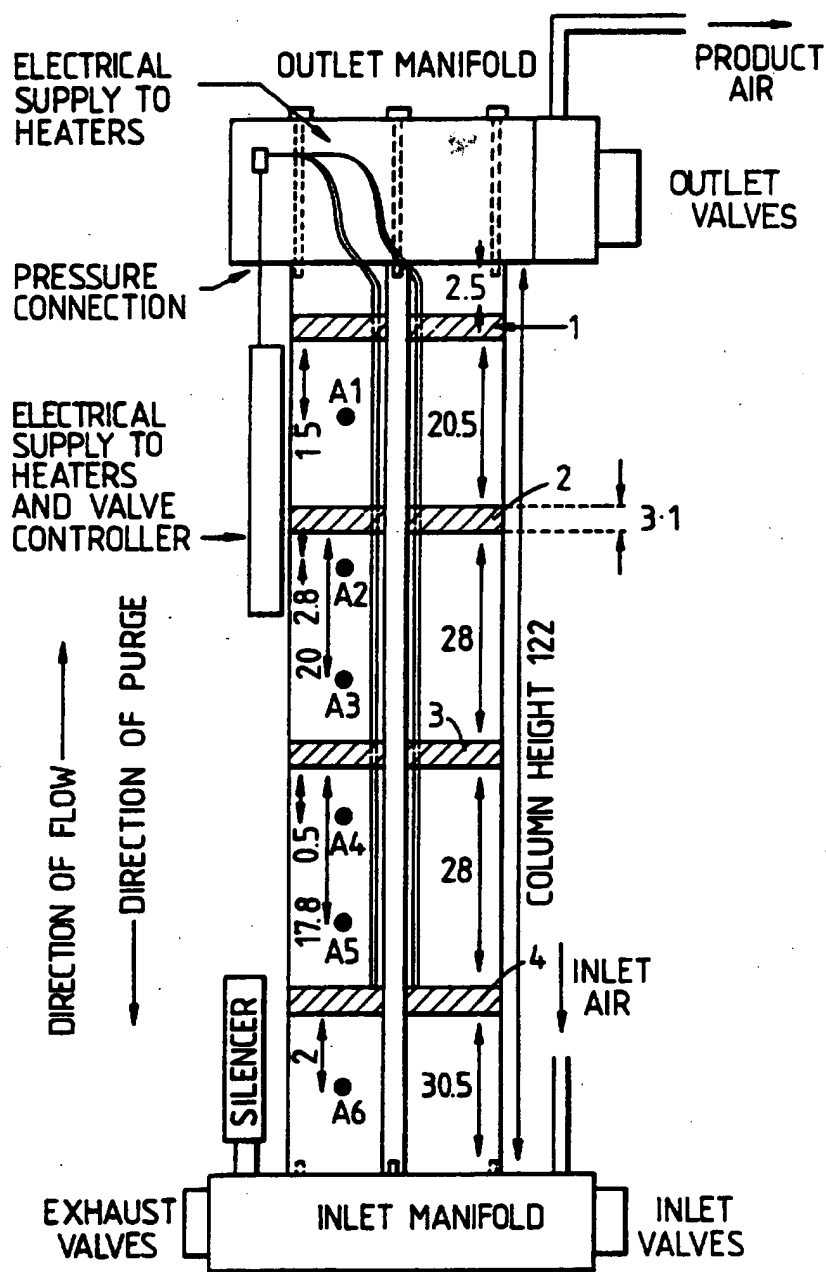


Fig.5b.



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Fig.6.



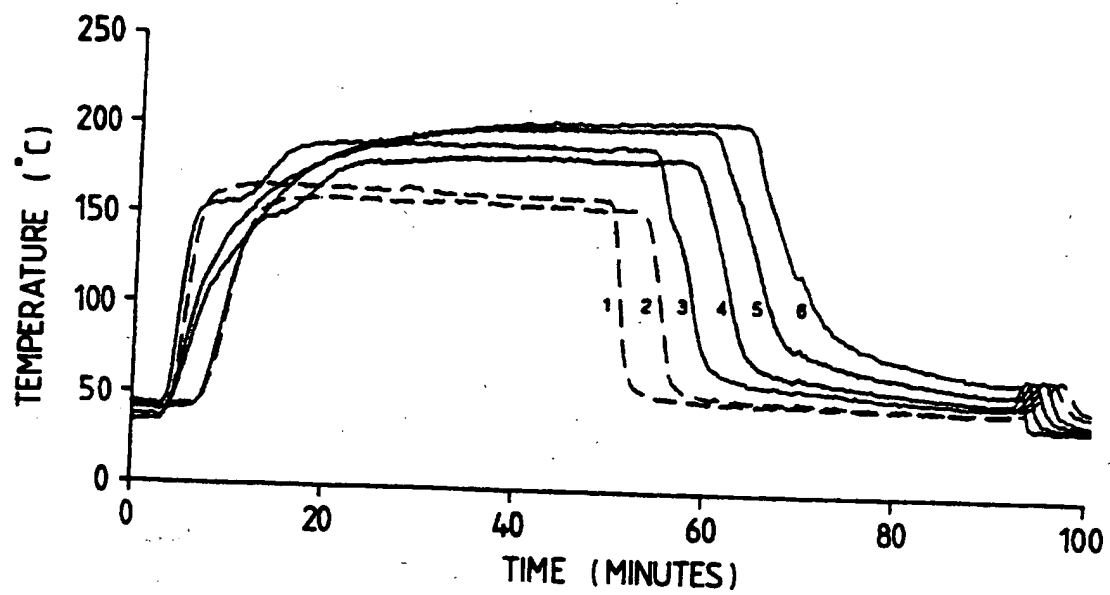
A1-A6 = THERMOCOUPLES

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Fig.7.

1-6= THERMOCOUPLES A1 - A6



INTERNATIONAL SEARCH REPORT

tional Application No
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A. CLASSIFICATION OF SUBJECT MATTER IPC 6 B01D53/04		
According to International Patent Classification (IPC) or to both national classification and IPC		
B. FIELDS SEARCHED Minimum documentation searched (classification system followed by classification symbols) IPC 6 B01D		
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Electronic data base consulted during the international search (name of data base and, where practical, search terms used)		
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Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US,A,3 193 985 (G. K. A. SIGGELIN) 13 July 1965 see column 3, line 19 - column 4, line 58; figure 1 ---	1,2,7
A	DE,C,549 531 (METALLGESELLSCHAFT) 14 April 1932 see page 2, line 61 - page 3, line 59; figures -----	1-5,7
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Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US-A-3193985	13-07-65	NONE	
DE-C-549531		NONE	